#### INTRODUCTION TO SPACE PROCESSING

By

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In organizing this symposium we had two purposes in mind: one is to present to you experiment results: that is the results of Skylab and of other experiments; the other purpose which may be even more important is to show you opportunities for building on the existing basis of experience for the profitable exploitation of the space environment.

We held Space Processing Symposia in 1968 and 1969. We said then that space processing would yield scientifically useful knowledge and commercially useful products. We also invited broad participation in the program. By necessity, the papers presented contained mainly projections on the advantages of processing in space new or improved materials for use on earth. For processing materials the unique and most important aspect of space flight is that it provides an environment effectively free from the influence of gravity. We postulated that in the absence of gravity effects the theoretical properties of materials could be more nearly approached because thermal convection would not disturb the even progression of solidification in crystals and eutectics, because immiscible components or phases of differing densities would not separate, and because, by containerless processing, contamination of a molten material by the crucible would be prevented; we speculated on many other promising areas of endeavor, including the purification and separation of biologicals.

Since these early days of brain storming we have made progress. By progress I mean that we are in a position to continue accelerating our efforts from a now existing foundation of experimental knowledge and of world-wide participation.

At the time of our early symposia we could predict no specific space flight opportunities. But now we have assurance of flight opportunities because the Space Shuttle program has become a reality. The thrust to avail ourselves of the payload capability of the Shuttle lends urgency,

direction and purpose to our endeavors. The Shuttle will fly in five to six years and before then we have to develop the technologies and perform precursor tests so that we will be able to take every advantage of the Space Shuttle capabilities for commercial and scientific goals.

In my talk, I want to give you an overview of the activities during the past few years. Many details will be filled in by the papers to be given in this symposium. It will become apparent that the materials and apparatus technologies have grown step by step: indeed in most instances it is possible to trace the development of today's most advanced concepts to early experiments. I will conclude by describing the Skylab materials processing facility in which the Skylab experiments were performed. This description will lead into the subject for today -- Skylab Results.

Figure 1 shows the accelerating pace of space processing activities. You will note that the number of planned studies has been increasing year by year; this means that the program has broadened not only in technical content but also in participation by industry, universities and government. Five Scientific Advisory Groups in various disciplines have been established; further groups in other discipline areas will be established as necessary. The advisory groups have been and continue to be most helpful in defining the study efforts necessary to advance the scientific disciplines, and in the evaluation of the results.

The technical studies which were performed both by universities and industry have had various objectives — first of all to provide a theoretical base for the understanding of the behavior of materials and the interaction of solids, liquids and gases with each other and with their environment when the effective gravity level is reduced by several orders of magnitude. Then other studies were to provide theoretical data on materials of industrial or scientific interest and finally the studies were to provide the design of ground based tests to validate certain assumptions or to verify study results. While laboratory one 'g' tests were sometimes helpful, in general low 'g' tests were required for this purpose.

# LOW 'G' TEST CAPABILITIES

We have developed several methods of low 'g' testing. The most accessible tests are laboratory tests where under certain circumstances low 'g' times of the order of one second may be attained. The next most accessible facility is the drop tower (Figure 2). A free-fall time of about four seconds is attainable and otherwise this device has few limitations. A surprising amount of data can be obtained in this apparently short period.

The experimental KC-135 aircraft flying Keplerian trajectories provides 10 - 20 seconds of low 'g'. It was used to verify the functioning of two of the Skylab experiments: Metals Melting (M551) and Sphere Forming (M553). This however within the limitation of the prevailing 'g' levels which were fluctuating and of the order of 0.01 'g'. Because of these unacceptable fluctuations, this method of operation for low 'g' experimentation has been abandoned.

Another method of obtaining a low 'g' environment is to use the free coast phase of a sounding rocket flight; i.e., the time when the rocket is above the atmosphere and no propulsion is applied. Typically four to seven minutes of low 'g' time is attainable. A beginning has been made toward a test program using such rockets.

These ground based tests of course are quite limited in duration and are regarded as precursors to more elaborate space flight experiments.

In space flight we have flown demonstrations on Apollo 14, 16, and 17, and of course experiments on Skylab. On these flights, experiment times were long and the 'g' levels were generally very low, although as you will hear, attitude control thrusters, equipment operation, gravity gradient and crew movements sometimes have a noticeable influence.

Figure 3 shows a summary chart of tests performed. I will take a few examples in each category to illustrate the importance of this test effort.

# MATERIALS

In the field of immiscibles, that is, compositions of materials having a miscibility gap in the liquid phase, the first demonstration of the existence of a stable dispersion in bulk samples was performed on Apollo 14 with Paraffin and Sodium Acetate (Figure 4); subsequent tests with a variety of metallic mixtures were performed on aircraft and in the drop tower. From these tests bulk samples with novel structures and properties were obtained. A single Ga-Bi sample (Figure 5) processed in the drop tower, for example, showed semi-conducting, conducting, and super-conducting properties. The results obtained led to the Skylab experiments which you will hear described later. In the case of immiscibles the progression from model materials to useful materials is well along; however, many new compositions and variations of processing parameters remain to be studied and tried. Over 500 materials compositions in this category have been identified many of which will need to be studied by processing in low 'g'.

For the growth of single crystals and eutectics relatively much longer processing times are needed. InBi cyrstals and eutectics were to be grown on Apollo 14 (Figure 6); however, crew time did not permit the performance of these particular experiments. Nonetheless, the experience gained from development and one 'g' ground testing contributed to the success of several Skylab crystal growth experiments.

The eleven materials samples that were processed on Apollo 14 included fiber and particle reinforced composites. These demonstrations indicated that homogeneous distributions of reinforcements can be attained and that wetting of the reinforcement by the matrix material is an important factor. Substantial differences in density between the matrix and the reinforcements had no effect on the distribution. This conclusion appears to be confirmed by one Skylab experiment, Whisker Reinforced Composites (M561).

## PROCESS ELEMENTS

In order to produce materials the individual factors influencing the processes in the absence of gravity forces have to be understood. For this reason, experimental studies on heat flow and convection in a low gravity environment were performed on Apollo 14 and 17. It had been postulated that in such an environment, thermal convection currents would not occur; heat transfer would occur only by conduction, radiation, and by surface tension driven convection. Figure 7 shows the apparatus. In these Apollo demonstrations, a liquid having a free surface did in fact exhibit the predicted surface tension driven convection seen as Benard cells (Figure 8). On Apollo 14, the contained liquids and gases exhibited small though significant amounts of steady state as well as oscillating convection. On Apollo 17, these demonstrations with slight modifications were repeated. From studies of the data from both flights, it was concluded that the convection observed on Apollo 14 resulted from accelerations exceeding 10<sup>-4</sup> 'g' caused by spacecraft or apparatus movement; on Apollo 17, because of appropriate precautions, accelerations were reduced to below  $10^{-6}$  'g' and similar effects were not observed. An important conclusion is that under low 'g' conditions in space thermal convection effects can be eliminated or controlled. The results of these preliminary tests appear to be confirmed by the Skylab experiments; particularly by the Radioactive Tracer Diffusion experiment (M558) and by several crystal growth experiments.

Another factor influencing the behavior of materials is wetting, for example wetting of the container by the contained material. One important discovery was made during the evaluation of a short duration rocket flight: on earth, contact is made between a fluid and its container by gravity; consequently,

heat can be readily extracted through the container whether the fluid wets the container or not. In the rocket flight we discovered the cooling and solidification of a molten InBi alloy contained in an aluminum cartridge did not proceed as fast as predicted from ground tests and some material remained molten on reentry. In the Apollo 14 InBi samples unusual solidification patterns were observed. Both effects were attributed to the fact that the melt did not wet the cartridge wall and only touched it at random spots. The melt, in effect, floated free within the cartridge.

The influence of contact with the container wall on the solidification characteristics of the molten material still has many unknowns particularly during processes as sensitive as the growth of crystals.

Another wetting effect was observed where a mixture of molten materials present: preferential wetting of the wall by one material was a mechanism for segregation (Figure 4).

# PROCESSING SYSTEMS

Various systems have been developed for processing materials in low 'g'. I want first to mention electrophoretic separation devices.

Electrophoresis in a free solution was first demonstrated on Apollo 14. While problems to do with apparatus design and sample materials were encountered, the results in a cell containing two different dyes indicated that the effort was worth pursuing. For Apollo 16, the mechanical equipment problems were solved and different model materials for separation were chosen. The apparatus is shown in Figure 9. During performance of the demonstration, back flow along the walls occurred owing to electro-osmosis. This distorted the separation fronts (Figure 10). However, the space demonstration showed that the deleterious effects due to gravity induced sedimentation and thermal convection were eliminated in low 'g'. The next experiments in this field are planned for the Apollo Soyuz mission (ASTP).

Systems for the containerless positioning of materials may be based for example on acoustic or on electromagnetic principles. For laboratory tests on glasses levitation in an airstream and location of the specimen on a sting have been used.

The value of drop tower tests was shown again during the development of an electromagnetic positioning system. Initial tests were performed on the ground: a test mass was suspended within the working volume of the system

# SKYLAB EXPERIMENT FACILITY AND MODULES

Finally, I want to describe the M512 processing facility in which all Skylab space processing experiments were performed. It was mounted in the Multiple Docking Adapter of the Skylab (Figure 15). The facility itself is shown in the next slide (Figure 16).

The M512 facility provided the capability of performing the experiments by the use of individual experiment modules.

The facility was mounted on a honeycomb panel which in turn was attached to the MDA structure by shock mounts. The panel also served as a radiator of heat.

The facility was made up of the following main parts:

- o Work chamber
- o Control panel
- Electron beam gun and batteries
- Storage boxes for experiment modules and ancillary equipment

The work chamber incorporated a mount to accommodate each experiment module in turn; the mount doubled as a heat sink with a predetermined and calibrated thermal impedance. Near the mount and inside the chamber was located an electrical connector through which power and signals were provided to all the experiment modules except the Multipurpose Furnace, which was a late addition and used only the heat sink/mount and the vacuum capability of the chamber.

The work chamber could be vented to space and thus provide an evacuated environment for the experiments when desired. Windows and illumination within the work chamber permitted viewing of the experiments in progress and also photography by a fixed camera. Gages on the control panel (Figure 17) served to monitor the pressure in the chamber, certain temperatures and the beam current and voltage of the electron beam gun. In addition, switches and potentiometers served to operate and control the individual experiments.

The Metals Melting (M551) and Sphere Forming (M553) experiments used the electron beam gun to melt the specimens. It was a most compact gun for its 2 KW output (100mA at 20,000 V). The power supply package was contained in a canister pressurized with an insulating gas: perfluoropropane ( $C_3F_8$ ). The filament and focusing devices were exposed to the vacuum of the work chamber.

The gun was powered by batteries and thus was independent of the spacecraft power bus. Incidentally, the M552 Exothermic Heating experiment also derived its igniter power from these same batteries. All other experiments operated on spacecraft power.

Most of the experiment modules and their accessories were stored in the storage container (Figure 18). To perform an experiment the appropriate module with its accessories was mounted in the chamber.

For the performance of the Metals Melting (M551) experiment the discs (Figure 19) were driven by a motor. The electron beam was focused on a tungsten target incorporated in each disc; the disc was then rotated at constant speed to make a weld bead and finally was stopped to melt a puddle. Three discs of different materials were provided for this experiment. They were processed successively.

The sphere forming specimens (M553) were carried on two wheels which were successively mounted on an indexing motor, each wheel carried a tungsten focusing target and 14 specimens. After each specimen was melted by the electron beam, a new specimen was indexed into position.

The Exothermic Brazing experiment container (M552) (Figure 20) held four exotherm packages. After the chamber was evacuated the exotherm packages were fired one at a time at about two hour intervals. The long intervals were necessary to allow for cooling between operations.

A set of experiments on Flammability (M479) was also performed in the chamber. To perform these experiments, an adapter was mounted in the chamber on the experiment connector; each specimen in turn was mounted in the adapter and ignited. The specimens could be burnt freely, or the combustion could be quenched by evacuating the chamber. Another quench system, by water spray, was also provided.

The Multipurpose Furnace System (M518) (Figure 21) consisted of the furnace, a control package and interconnecting cabling.

To perform the eleven experiments (M556 through M566), the furnace was installed in the work chamber, the control package was mounted on its special adapter and interconnections were made from the controls to the furnace, to the Skylab power receptacle and to the Skylab telemetry system. After the insertion of each set of three cartridges for an experiment, the work chamber was evacuated and the experiment was initiated after setting the appropriate peak temperature, soak time and cool-down rate. Lights indicated

what particular period of the total cycle had been reached. Thermocouples at the "hot" and "cold" ends of the cartridges provided outputs which were telemetered to the ground for real time evaluation of the progress of the experiments.

# CONCLUSION

In this talk I have shown with a few examples that the space processing program is structured to allow progress from studies through ground based low 'g' tests to space flight experiments. This progression should and will go further. It is in this sense that our symposium is intended not as a monument to past achievements but as a stepping stone to future applications and benefits.

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FISCAL YEAR	1969	1970	1971	1972	1973	1974
STUDIES PLANNED	9	12	20	25	47	68
LOW 'G' TESTS						
DROP TOWER	-	ı	9	64	70	144
RESEARCH AIRCRAFT	i		3	2	10	
ROCKET FLIGHTS			<b>A</b>	<u> </u>	,	<b>A</b>
APOLLO			14	. 🛕	<b>A</b>	
SKYLAB			14	16	17	
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FIGURE 1. SPACE PROCESSING ACTIVITIES

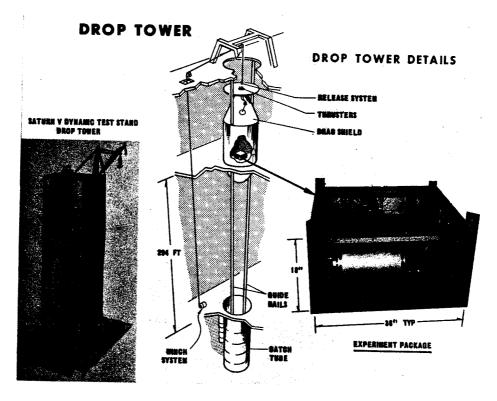


FIGURE 2. LOW "G" DROP TOWER FACILITY

MATERIAL  CRYSTALS  IMMISCIBLES  COMPOSITES  GLASSES  BIOLOGICALS  PROCESS ELEMENTS  HEAT FLOW/CONNECTION  WETTING  PROCESSING SYSTEMS  HEATING/COOLING  CONTAINERLESS POSITIONING  HEATING/COOLING  CONTAINERLESS POSITIONING  HOMOGENIZATION/DISPERSION  SEPARATION/PURFICATION  A  A  A  A  A  A  A  A  B  B  B  B  B				3. 100 00 00 00 00 00 00 00 00 00 00 00 00	( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )	Age Carry	A A STORY	20 4 20 4	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\		20/2	
CRYSTALS         A         B         A         B           IMMISCIBLES         A         A         A         A         B           COMPOSITES         A         A         A         A         A         B         B         B         A	MATERIAL	<u> </u>	<u>/\§</u>	<u> </u>	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	<u> </u>	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	1/8	1/6	1/3	1/4	7
IMMISCIBLES         △ <t< td=""><td></td><td><b>A</b></td><td>[</td><td></td><td></td><td>•</td><td></td><td></td><td>Ī</td><td><b>A</b></td><td>•</td><td>1</td></t<>		<b>A</b>	[			•			Ī	<b>A</b>	•	1
COMPOSITES         △ <td< td=""><td></td><td>_</td><td></td><td><b>A</b></td><td><b>A</b></td><td><b>A</b></td><td></td><td></td><td></td><td>•</td><td>•</td><td>1</td></td<>		_		<b>A</b>	<b>A</b>	<b>A</b>				•	•	1
GLASSES BIOLOGICALS  PROCESS ELEMENTS  HEAT FLOW/CONNECTION  DIFFUSION  WETTING  PROCESSING SYSTEMS  HEATING/COOLING  CONTAINERLESS POSITIONING  HOMOGENIZATION/DISPERSION  A  A  A  A  A  A  A  A  A  A  A  A  A		. 🛦				<b>A</b>			<b>A</b>	<b>A</b>		
PROCESS ELEMENTS  HEAT FLOW/CONNECTION	EUTECTICS	<b>A</b>				•				<b>A</b>		
PROCESS ELEMENTS  HEAT FLOW/CONNECTION	GLASSES	<b>A</b>	•									}
HEAT FLOW/CONNECTION A A A A A A A A A A A A A A A A A A A	BIOLOGICALS	•				<b>A</b>						}
DIFFUSION  WETTING  A  A  A  A  A  A  B  PROCESSING SYSTEMS  HEATING/COOLING  CONTAINERLESS POSITIONING  HOMOGENIZATION/DISPERSION  A  A  A  A  A  A  A  A  A  A  A  A  A	PROCESS ELEMENTS											
PROCESSING SYSTEMS  HEATING/COOLING CONTAINERLESS POSITIONING HOMOGENIZATION/DISPERSION  A A A A A A A A A A A A A A A A A A A	HEAT FLOW/CONNECTION	<b>A</b>				<b>A</b>		A				]
PROCESSING SYSTEMS  HEATING/COOLING  CONTAINERLESS POSITIONING  HOMOGENIZATION/DISPERSION  A  A  A  A  A  A  A  A  A  A  A  A  A	DIFFUSION	•		Ĺ						<b>A</b>		
HEATING/COOLING  CONTAINERLESS POSITIONING  HOMOGENIZATION/DISPERSION  A  A  A  A  A  A  A  A  A  A  A  A  A	WETTING	<b>A</b>		<b>A</b>	<b>A</b>	•			<b>A</b>	<b>A</b>		
CONTAINERLESS POSITIONING A A A B A A B A A B A B A B A B A B A	PROCESSING SYSTEMS											_
HOMOGENIZATION/DISPERSION A A A		•		<b>A</b>	<b>A</b>	<b>A</b>			<b>A</b>	<b>A</b>		]
		<b>A</b>		<b>A</b>	_	•		L		<b>A</b>		1
SEPARATION/PURFICATION   A     A   A   B					<b>A</b>	<b>A</b>	<u> </u>		<u> </u>	_		
	SEPARATION/PURFICATION	<b>A</b>	<u> </u>		L		_	<u> </u>	L			J

- ▲ TESTS PERFORMED.
- TESTS FLOWN BUT NOT PERFORMED
- = ASTP PLANNED

FIGURE 3. SPACE PROCESSING - TESTS AND EXPERIMENTS

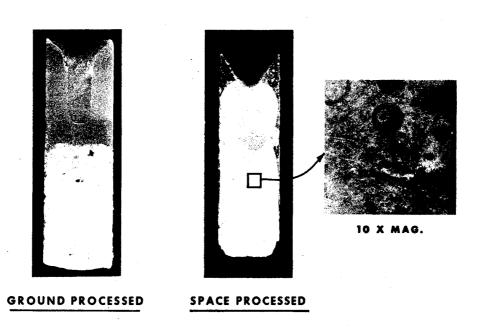
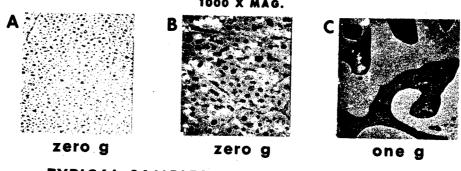
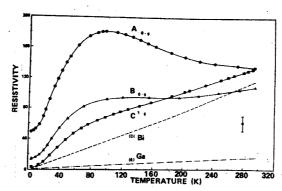


FIGURE 4. APOLLO 14 - IMMISCIBLE MATERIALS PROCESSING, SODIUM ACETATE - PARAFFIN



TYPICAL SAMPLES OF IMMISCIBLES Ga-Bi



# **ELECTRICAL RESISTIVITY OF SAMPLES**

FIGURE 5. TYPICAL RESULTS OF IMMESCIBLES Ga-Bi PROCESSED IN THE DROP TOWER

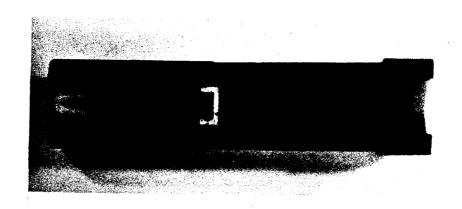


FIGURE 6. BRIDGEMAN CRYSTAL CARTRIDGE

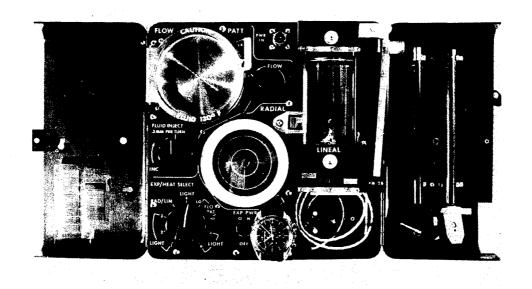


FIGURE 7. APOLLO 17 HEAT FLOW AND CONVECTION DEMONSTRATION (RECORDING CAMERA OMITTED)

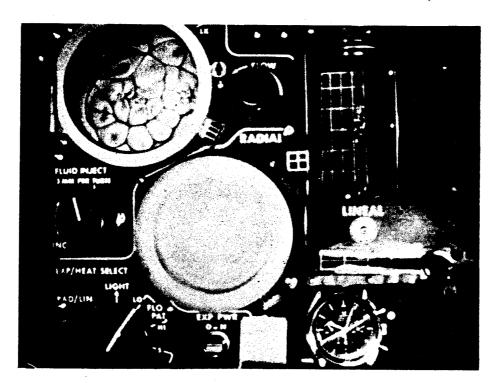


FIGURE 8. APOLLO 17 HEAT FLOW AND CONVECTION DEMONSTRATION: SURFACE TENSION DRIVEN CONVECTION IN LOW-G (BENARD CELL)

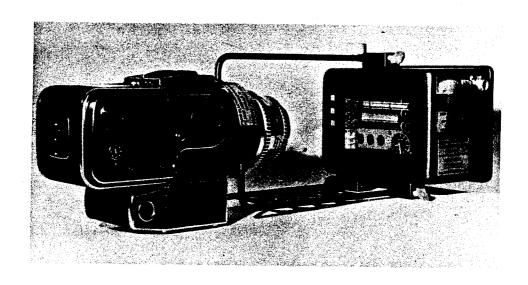


FIGURE 9. APOLLO 16 ELECTROPHORESIS DEMONSTRATION

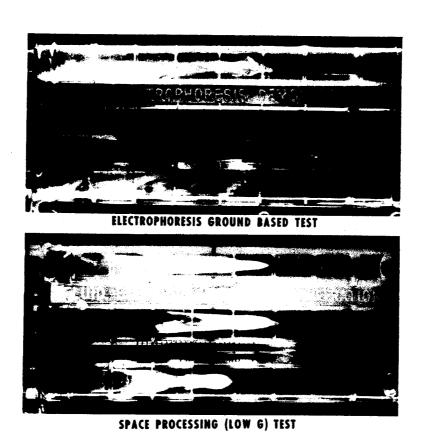


FIGURE 10. COMPARISON OF RESULTS OF GROUND BASED AND APOLLO 16 ELECTROPHORESIS DEMONSTRATION

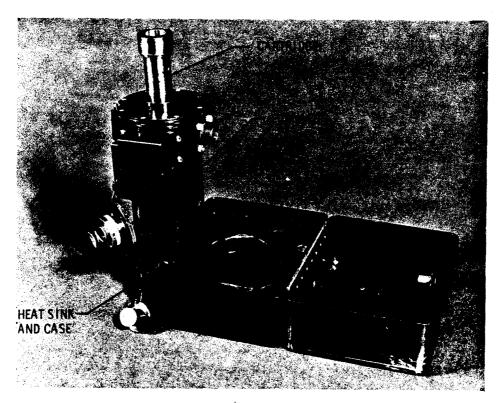


FIGURE 11. APOLLO 14 FURNACE FOR CASTING DEMONSTRATION

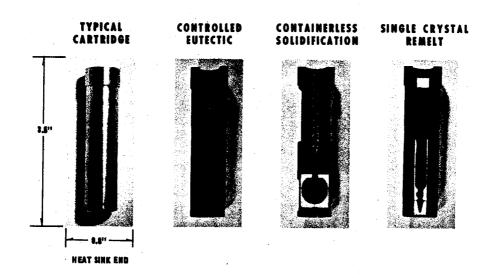


FIGURE 12. APOLLO 14 CARTRIDGES FOR CASTING DEMONSTRATION (INDIUM BISMUTH)

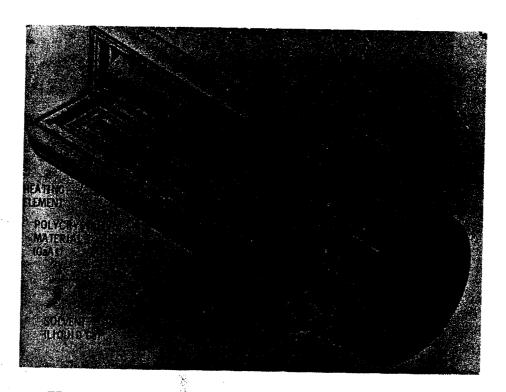


FIGURE 13. M555 - SINGLE CRYSTAL GROWTH EXPERIMENT

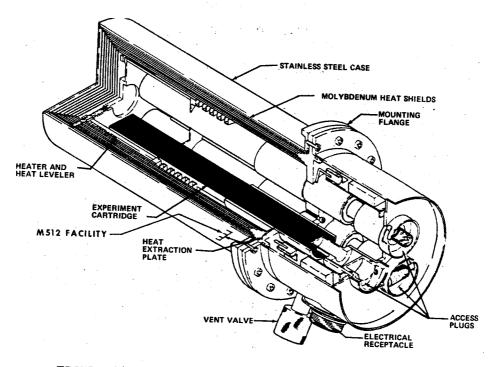


FIGURE 14. M518 - MULTIPURPOSE ELECTRIC FURNACE

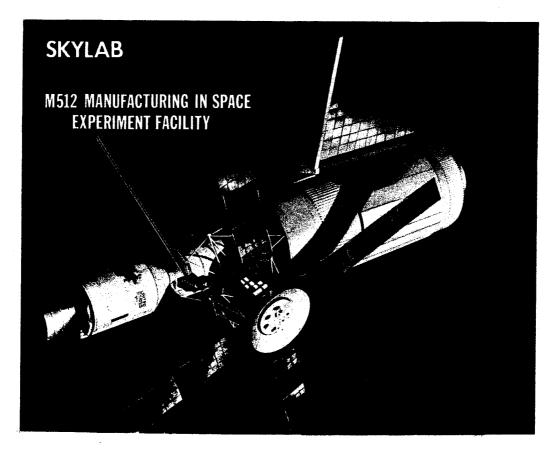


FIGURE 15. LOCATION OF M512 EXPERIMENT ON SKYLAB



FIGURE 16. SKYLAB M512 MATERIALS PROCESSING IN SPACE EXPERIMENT FACILITY



FIGURE 17. M512 EXPERIMENT CONTROL PANEL

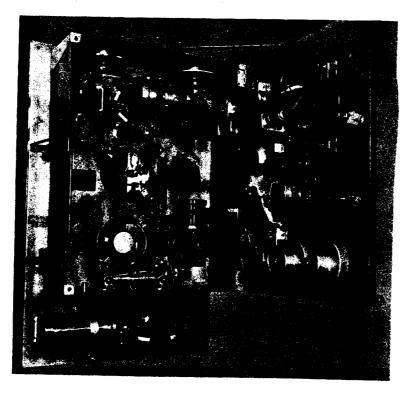


FIGURE 18. M512 EXPERIMENT ACCESSORIES STOWAGE CONTAINER





M551 METALS MELTING EXPERIMENT

M553 SPHERE FORMING EXPERIMENT

FIGURE 19. M551 METALS MELTING EXPERIMENT AND M553 SPHERE FORMING EXPERIMENT



M552 EXOTHERMIC HEATING
EXPERIMENT



M479 FLAMMABILITY EXPERIMENT

FIGURE 20. M552 EXOTHERMIC HEATING EXPERIMENT AND M479 FLAMMABILITY EXPERIMENT

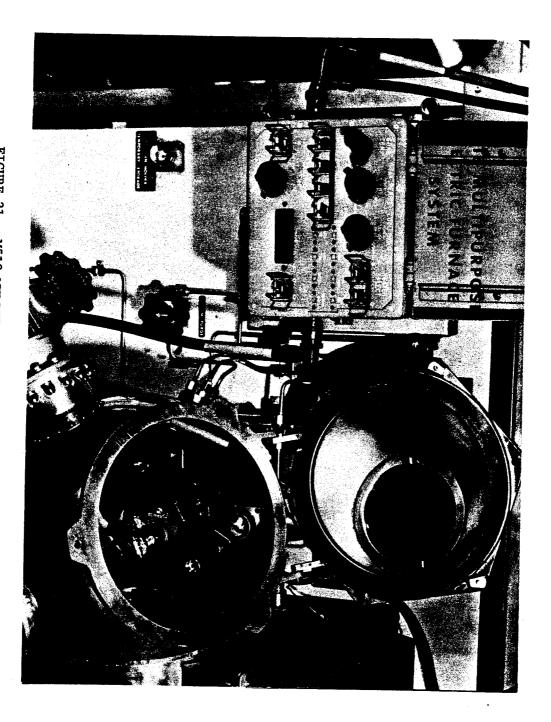


FIGURE 21. M518 MULTIPURPOSE ELECTRIC FURNACE SYSTEM

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